Nuclear Instruments and Methods in Physics Research B 365 (2015) 175–181

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

The influence of the ion beam on the structure and optical properties of titanium nitride nano-scale thin films

Ibrahim Odeh [*], Rajaa Elian

Physics Department, Yarmouk University, Irbid 21163, Jordan

ARTICLE INFO

Article history:
Received 1 October 2014
Received in revised form 30 April 2015
Accepted 4 August 2015
Available online 17 August 2015

Keywords:
Ion beam assisted deposition
Titanium nitride
Nano thin films
Optical constants
Decorative coating

ABSTRACT

Titanium nitride nano-scale thin films have been prepared by ion beam assisted reactive DC magnetron sputtering. The films are characterized by XRD, SEM and TEM. The films are found to be amorphous. The effect of the ion beam during deposition was evident from smoothness of film surface (SEM and TEM images) and modifications in optical properties. Investigation of the optical constants shows stable refractive index dominating most of the visible range. The films are not highly absorptive in the visible range. An energy gap of 2.9 ± 0.1 eV is estimated for the IBAD amorphous titanium nitride nano-thin films. The stability of the films at normal room environment in addition to the golden color makes the nano-thin films suitable for hard and decorative coatings.

1. Introduction

Titanium nitride (TiN) is a well-known hard coating material. Thin films of TiN deposited by physical vapor deposition PVD are used industrially on tools. Films deposited by Chemical Vapor Deposition CVD and PVD have improved tribological properties of mechanical components, cutting tools. In addition, they are used for decorative purposes. CVD and PVD deposition processes promote the growth of the columnar structure however [1,2].

Titanium nitride films have high hardness, low friction coefficient and high temperature resistance. They strongly adhere to several types of substrates [3]. In addition, coated cutting tools with TiN films show improved machining performance and wear and corrosion resistance [4,5]. Thin films of titanium nitride exhibit an attractive appearance due to their golden color. TiN coatings prepared by PVD techniques exhibit good corrosion resistance and an attractive gold-like appearance. Titanium nitride (TiN) thin films are widely used as adhesion layers, diffusion barriers in device interconnects [6] also as a direct-metal-gate material for metal–oxide–semiconductor devices due to the thermal stability and low bulk electrical resistivity of TiN [7,8]. With device dimensions constantly decreasing, the required film thickness is approaching a few nanometers. For such thicknesses the continuity of a film becomes an important factor [9–11].

Ion bombardment can significantly modify thin film growth and surface morphology during the deposition. It has shown to increase the film density, inhibit the formation of a columnar structure and decreases the average grain size.

Films produced by ion beam deposition and ion beam assisted deposition have several advantages in thin film applications. Compared with thermal energy processes, such as evaporative deposition, the beam ions are more energetic. Compared with plasma processes, ion sources permit independent control of ion energy, ion density, direction and background pressure.

In general, ion sources can be operated at low background pressures and therefore are suited for processes requiring low contamination from background impurities and low temperature. Ion beam assisted deposition (IBAD) is generally used in conjunction with other types of thin film deposition methods. IBAD enhances the properties of the film without the requirement for additional heating of the substrate.

The most dramatic effects of ion assist are in film densification and the reduction of film stress and columnar structure. This enables the user to eliminate the need for additional substrate heating.

Additional enhancements in film quality have been shown in control of film microstructure, orientation and temporal environmental stability. The use of ion beam assists greatly in the control of proper film stoichiometry, particularly for oxides, and nitrides [12–17].

Because of the importance of the TiN material, the present study aimed at firstly, prepare nano scale thin films by ion beam

---

[*] Corresponding author.
E-mail address: iodeh@yu.edu.jo (I. Odeh).

http://dx.doi.org/10.1016/j.nimb.2015.08.007
0168-583X/

assisted reactive DC magnetron sputtering. Secondly, characterize and study the optical properties of the nano-scale thin films and the effect of ion bombardment on these properties during deposition.

The novelty of the work described here stems from the nano scale thicknesses of the films and the simultaneous use of DC magnetron reactive sputtering and ion beam assisted deposition (IBAD). Most of the work reported in the literature was on thicker TiN films produced by thermal or electron beam deposition and IBAD.

2. Experimental and methods

2.1. Deposition system set-up and preparation of titanium nitride nano thin films

In this work we have employed the reactive DC sputtering technique and the ion beam assisted deposition (IBAD) simultaneously to prepare titanium nitride films. Fig. 1 shows the actual setup and vacuum chamber during sample deposition. We have used an End-Hall type ion beam source specifically designed, built, and retrofitted to the vacuum chamber in such a way that ions from source were directed to imping onto the substrate at about 50–55° during deposition.

A low-tension high-current transformer provided the necessary power to the W-filament of the ion source during deposition. An independent power supply (HP 6521A) provided the accelerating voltages of 200–300 V applied to the ion source such that the ions leaving the source would have energy of about 100–150 eV. We have not been able to measure the ion current but we roughly think it was in region of 1–3 A.

A 75 mm DC magnetron sputtering accessory (Edwards, UK) fitted with a high purity Titanium target formed the main source of titanium atoms to react with the Nitrogen plasma. A novel substrate holder (Rotating Hexa-holder) capable of loading six substrates simultaneously was designed and constructed specifically for this type of sample deposition as shown in Fig. 1. The distance between the glass substrate and the source (Ti) target was kept at about 10 cm.

A 1 kW DC power supply (MDX 1 K Magnetron Drive, Advanced Energy, USA) delivered the DC power to the water-cooled titanium target.

A thoroughly cleaned glass substrates were used throughout this work. In addition to normal cleaning the glass substrates were bombarded with the ion source for five minutes prior to deposition to remove any traces from the surface.

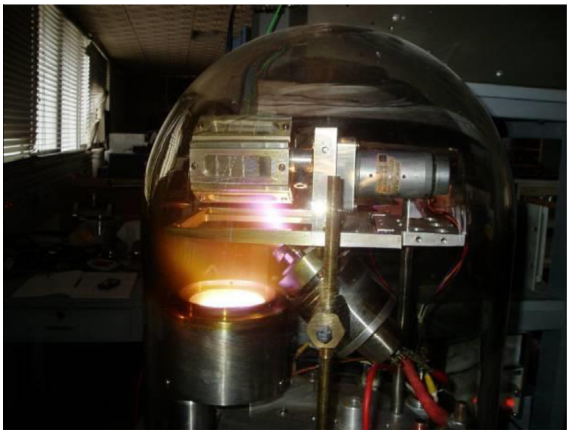

Fig. 1. The DC magnetron sputtering system with ion beam source in operation during deposition. In addition, shown is the sample-holder (Rotating Hexa-holder).

The following is a typical sample preparation procedure:

The vacuum chamber was evacuated to base pressure of better than (2×10^{-5}) mbar. The reactive gas, nitrogen, was introduced to the chamber through a controlled needle valve until a working pressure of (2×10^{-2}) mbar was reached. A 380–400 W DC power was delivered to the Titanium target. This amount of power was found to be sufficient to sustain the plasma and produce a reasonable deposition rate. The substrate holder was kept at a substrate to target distance of 10 cm. The holder was kept at ground hence the substrate. This distance was found to minimize thickness variations during deposition. The holder rotates at controlled speeds in such a fashion that the substrates always face the sputtering target one at a time during deposition. The duration of the deposition was anywhere from 10 to 20 min. We have attempted to measure the film thickness by using a Quartz Crystal Monitor. This did not yield reliable results due some technical problems. Instead we have determined the thicknesses from the optical transmittances by employing PUMA software used in the determination of optical constants [18–20]. The transmittance T in the wavelength range between 360 and 900 nm was recorded using a UV–Visible double-beam spectrophotometer.

3. Experimental results

3.1. XRD spectrum

Fig. 2 shows a typical XRD spectrum representative of titanium nitride films prepared and studied in this work.

It is clear from the figure that the spectrum does not exhibit any diffraction peaks which are typical of the crystalline structure. This lack of peaks is consistent with amorphous structure.

3.2. Transmission electron microscopy (TEM)

The images captured from the (TEM) show that the films are flaky and without any crystalline shapes suggesting amorphous titanium nitride thin films which supports the results obtained by XRD. They also show a rather loose structure and what might be seen as larger grain sizes. In addition, there is a clear difference between the images obtained for samples prepared by dc magnetron sputtering without using the ion beam source, Fig. 3, and those samples prepared using ion beam source, Fig. 4. In the IBAD films the nano size grains are clearly visible compared those films prepared without IBAD.

The ion beam source substantially improved properties of the films and contributed to the increase in the film density. It decreased the average grain size and inhibited the formation of a columnar structure as can be clearly shown in TEM image, Fig. 4 below.

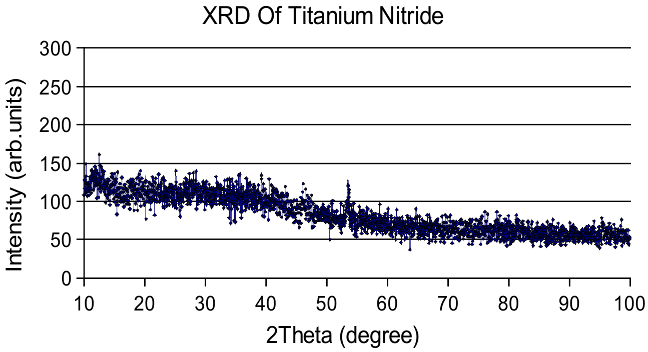

Fig. 2. A typical XRD spectrum of TiN films prepared ion this work indicating the amorphous nature of the samples.